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IS 6890-2 (1975): Methods for Chemical Analysis of Dental Gold Alloys, Part II: Determination of Nickel and Zinc [MTD 10: Precious Metals]



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IS : 6890 (Part II) - 1975

(Reaffirmed 2003)

Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF DENTAL GOLD ALLOYS PART II DETERMINATION OF NICKEL AND ZINC

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*Indian Standard*METHODS FOR CHEMICAL
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PART II DETERMINATION OF NICKEL AND ZINC

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Indian Standard

METHODS FOR CHEMICAL ANALYSIS OF DENTAL GOLD ALLOYS

PART II DETERMINATION OF NICKEL AND ZINC

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 19 May 1975, after the draft finalized by the Precious Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Dental gold alloys in the form of wire and castings, the compositions of which are covered in IS : 3578-1966*, IS : 4799-1968† and IS : 5954-1970‡ are used in dentistry for various purposes. These alloys contain besides gold, silver and platinum group metals, copper, nickel and zinc also. For accurate determination of its composition, the methods for analysis are covered in two parts of this standard. The determination of all elements except nickel and zinc are covered in Part I. This Part lays down the methods for determination of nickel and zinc in dental gold alloys.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960§.

1. SCOPE

1.1 This standard (Part II) prescribes the methods for determination of nickel and zinc (within the range 0 to 2 percent) in dental gold alloys covered in IS : 3578-1966*, IS : 4799-1968† and IS : 5954-1970‡.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960||) shall be employed in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for dental gold alloy wire.

†Specification for dental casting gold alloys.

‡Specification for dental white gold alloys.

§Rules for rounding off numerical values (*revised*).

||Specification for water, distilled quality (*revised*).

3. SAMPLING FOR CHEMICAL ANALYSIS

3.1 Sampling for chemical analysis shall be carried out in accordance with 4 of IS : 6890 (Part I)-1973*.

4. DETERMINATION OF NICKEL BY GRAVIMETRIC/PHOTOMETRIC METHOD BY DIMETHYLGLYOXIME

4.1 Outline of the Method — After removal of silver, gold, platinum, palladium and copper, nickel is precipitated by dimethylglyoxime, filtered, dried and weighed.

4.2 Reagents

4.2.1 Ammonium Chloride — solid.

4.2.2 Tartaric Acid Solution — Dissolve 250 g of tartaric acid in water and dilute to one litre.

4.2.3 Dimethylglyoxime Solution — Dissolve 10 g of dimethylglyoxime in one litre of rectified spirit (conforming to IS : 323-1959†). Filter before use.

4.2.4 Concentrated Ammonium Hydroxide Solution — $d = 0.90$.

4.2.5 Sodium Citrate Solution (100 g/l) — Dissolve 100 g of sodium citrate dihydrate in water. Dilute to one litre, and mix.

4.2.6 Dilute Ammonia Solution — 1 : 1 and 1 : 50 (v/v).

4.2.7 Chloroform

4.2.8 Dilute Hydrochloric Acid — 0.5 N.

4.2.9 Bromine Water — saturated.

4.2.10 Standard Nickel Solution — Dissolve 0.1000 g of nickel in 10 ml of dilute nitric acid (1 : 1). Boil to expel brown fumes, cool and dilute to one litre. Dilute 25 ml of this solution to 500 ml in a volumetric flask and mix. One millilitre of the solutions contains 0.005 mg of nickel.

4.3 Procedure

4.3.1 For Nickel More Than 0.5 Percent

4.3.1.1 Remove silver, gold, platinum, palladium and copper as specified in the methods given in IS : 6890 (Part I)-1973*. Boil the solution obtained under **9.4.3** of IS : 6890 (Part I)-1973* (after deposition of

*Methods for chemical analysis of dental gold alloys: Part I Determination of gold, silver, copper, palladium and platinum.

†Specification for rectified spirit (revised).

copper). Bring the solution to a volume of about 200 ml. Remove the beaker from the burner. Add about 2 g of ammonium chloride and 10 ml of tartaric acid solution and heat to about 80°C. Add 0.4 ml of the dimethylglyoxime solution for each milligram of nickel present in the solution; and 5 to 10 ml in excess, pouring directly into the solution and not down the beaker wall. Neutralize the solution with concentrated ammonium hydroxide solution, stirring vigorously and add 1 ml in excess. Allow to cool to room temperature while stirring occasionally. Allow the precipitate to settle for 2 hours on water-bath.

4.3.1.2 Filter the precipitate on a weighed sintered glass crucible (G3) and wash with cold water till free of chloride. Reserve the filtrate for determination of zinc.

4.3.1.3 Dry the precipitate obtained under **4.3.1.2** at 120°C to constant mass. Cool in a desiccator and weigh as nickel-dimethylglyoxime.

4.3.2 *For Nickel Content ≤ 0.5 Percent*

4.3.2.1 Remove silver, gold, platinum, palladium and copper as specified in the methods given in IS : 6890 (Part I)-1973* and dilute the filtrate reserved under **9.4.3** of IS : 6890 (Part I)-1973* to a suitable volume. Take a suitable aliquot containing less than 5 micrograms of nickel in a separating funnel, add 5 ml of sodium citrate solution and make slightly ammoniacal and add a few drops of ammonia (1 : 1) in excess. Add 2 ml of dimethylglyoxime solution and extract with 2 to 3 portions of 5 ml chloroform, shaking for 1 minute each time.

4.3.2.2 Shake the combined chloroform extracts with 5 ml of dilute ammonia solution (1 : 50) and run the chloroform into another separating funnel. Shake the water layer with 1 to 2 ml of chloroform and combine the latter with the washed extract.

4.3.2.3 Shake the chloroform solution vigorously for 1 minute with two 5-ml portions of dilute hydrochloric acid. Transfer the hydrochloric acid solution to a 100-ml volumetric flask containing 50 ml of water.

4.3.2.4 Add 5 ml of bromine water and sufficient dilute ammonia solution (1 : 1), added dropwise, to just bleach the bromine, add 3 ml in excess. Cool and add 3 ml of the alcohol solution of dimethylglyoxime, taking care to complete the addition of bromine water, dilute ammonia solution and dimethylglyoxime solution within a period of 5 minutes, dilute to the mark and allow to stand for a definite period preferably 10 minutes after the addition of dimethylglyoxime solution.

4.3.3 Transfer a suitable portion of the solution to an absorption cell and measure the absorption at a wavelength of 530 nm. Run a blank using the same quantity of reagents.

*Methods for chemical analysis of dental gold alloys: Part I Determination of gold, silver, copper, palladium and platinum.

4.3.4 Calibration Curve — Take 0, 1, 2, 3, 4 and 5 ml of the standard nickel solutions into 100-ml volumetric flask containing 50 ml water, and proceed as under **4.3.2.4**. Record the photometric readings of all the standard solutions and draw a calibration curve by plotting the photometric readings of the standard solutions against milligrams of nickel.

4.4 Calculation — Convert the photometric readings of the sample to milligrams of nickel by means of the calibration curve and calculate the percentage of nickel as follows:

$$\text{Nickel, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of nickel in aliquot of the solution, and

B = mass in g of sample represented by the aliquot taken.

5. DETERMINATION OF ZINC BY THE DITHIZONE (PHOTOMETRIC METHOD) (FOR ZINC CONTENTS UP TO 0.5 PERCENT)

5.1 Outline of the Method — After separation of other elements, zinc is extracted in carbon tetrachloride solution of dithizone and intensity of the colour measured photometrically at 520 nm.

5.2 Reagents

5.2.1 Citric Acid Solution — 5 percent $\left(\frac{m}{v}\right)$.

5.2.2 Dithizone Solution — 0.001 percent in carbon tetrachloride. Prepare a concentrated solution and dilute when required. Purify the solution as under:

Dissolve the product in chloroform and treat it with ammonia. From the aqueous layer the pure dithizone is obtained as a precipitate on addition of mineral acid. The test for purity is that aqueous ammoniacal solution of dithizone should be colourless. Otherwise further purification is necessary.

5.2.3 Dilute Hydrochloric Acid — 0.2 N.

5.2.4 Acetate Buffer Solution (pH 4.7) — Dissolve 68 g of sodium acetate hydrate ($\text{NaC}_2\text{H}_3\text{O}_3$) and 29.4 ml of glacial acetic acid in water and dilute to 500 ml.

5.2.5 Sodium Thiosulphate Solution — Dissolve 25 g of crystalline sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 100 ml of water.

NOTE — Remove impurities from the reagents by shaking with 0.001 percent dithizone solution in carbon tetrachloride. Filter through dry filter paper to remove droplets of tetrachloride.

5.2.6 Standard Zinc Solution — Dissolve 0.1 g of pure zinc (99.95 percent purity) in 5 to 10 ml of dilute hydrochloric acid (1 : 1). Cool and dilute to one litre in a volumetric flask. Transfer 1 ml of this solution to 100-ml volumetric flask and make up the volume to the mark with water. One millilitre of the solution contains 1 μ g of zinc.

5.3 Procedure

5.3.1 Take a suitable aliquot (from the solution reserved under 4.3.1.2 or made up to a volume under 4.3.2.1 containing less than 5 μ g of zinc in a separating funnel. Add 5 ml of citric acid solution extract twice with 5 ml of dithizone solution, shaking vigorously for 2 minutes.

5.3.2 Mix the two extracts of the organic layers and back extract zinc with 5 ml of dilute hydrochloric acid. Discard the organic portion of the solution.

5.3.3 Add successively to the aqueous phase 5 ml of buffer solution, 1 ml of sodium thiosulphate solution. Shake vigorously for 2 minutes with 5 ml of dithizone solution. Dry the stem of the funnel with filter paper and run the clean carbon tetrachloride extract into 1-cm absorption cell and cover it.

5.3.4 Take the photometric reading of the solution at 520 nm. Run a blank using the same quantity of reagents.

5.3.5 Calibration Curve — Take 0, 1, 2, 3, 4 and 5 ml of standard zinc solutions and proceed as in 5.3.3. Record the photometric readings of all the standard solutions, and draw a calibration curve by plotting the photometric readings of the standard solutions against milligrams of zinc.

5.4 Calculation — Convert the photometric readings of the sample to milligrams of zinc by means of the calibration curve and calculate the percentage of zinc as follows:

$$\text{Zinc, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of zinc found in aliquot of the solution, and

B = mass in g of sample represented by the aliquot taken.

6. DETERMINATION OF ZINC BY EDTA METHOD (VOLUMETRIC) (FOR THE ZINC CONTENTS 0.5 TO 2 PERCENT)

6.1 Outline of the Method — After separation of other elements, zinc is precipitated by hydrogen sulphide gas, dissolved in hydrochloric acid and determined volumetrically by titrating with EDTA solution.

6.2 Reagents

6.2.1 *Acetic Acid* — glacial.

6.2.2 *Methyl Red Indicator* — Dissolve 1.0 g of the reagent in 60 ml of rectified spirit and dilute to 100 ml with water.

6.2.3 *Hydrogen Sulphide* — gas.

6.2.4 *Hydrogen Sulphide Wash Solution* — Saturate water with hydrogen sulphide gas.

6.2.5 *Dilute Hydrochloric Acid* — (1 : 1) (v/v).

6.2.6 *Potassium Cyanide Solution* (100 g/l) — Dissolve 100 g of potassium cyanide (KCN) in 500 ml of water and dilute to one litre.

6.2.7 *Buffer Solution* (pH 10) — Dissolve 54 g of ammonium chloride (NH_4Cl) in 300 ml of water, add 350 ml of ammonia and dilute to one litre.

6.2.8 *Eriochrome Black-T Indicator Solution* — Dissolve 0.4 g of eriochrome black-T in 20 ml of ethanol and 30 ml of triethanolamine and store in a polyethylene dropping bottle.

6.2.9 *Formaldehyde* (1 : 9) — Dilute 100 ml of formaldehyde (37 percent) with 900 ml of water.

6.2.10 *Disodium Ethylenediamine Tetraacetate (EDTA) Standard Solution* (0.01M) — Dissolve 3.7226 g disodium dihydrogen ethylenediamine tetraacetate in distilled water and dilute to one litre. Standardize the solution as follows:

Transfer to an 800-ml beaker an aliquot of the standard zinc solution approximately equal in zinc content to the aliquot of the sample and continue as per **6.3.3**.

6.3 Procedure

6.3.1 Acidify the filtrate reserved under **4.3.1.2** with glacial acetic acid using methyl red indicator. If the solution from the filtrate diluted to a volume reserved under **4.3.2.1** is to be used, pipette out a suitable quantity of the solution, precipitate nickel as nickel dimethylglyoxime as under **4.3.1.1**. Filter and acidify the filtrate with glacial acetic acid. Heat to boiling temperature and pass a rapid stream of hydrogen sulphide gas for about 20 to 30 minutes. Allow the zinc sulphide precipitate to settle and filter through a filter paper No. 1 using paper pulp. Wash the precipitate with hydrogen sulphide wash solution.

6.3.2 Transfer the paper along with the precipitate to the original beaker and add 20 ml of dilute hydrochloric acid. Heat to dissolve the precipitate, dilute to about 100 ml and boil to expel hydrogen sulphide gas. Cool, filter

and wash the paper thoroughly with hot water till free from acid. Transfer the solution to an 800-ml beaker.

6.3.3 Add 3 to 4 drops of methyl red indicator and carefully neutralize the solution with sodium hydroxide solution. Add 10 ml potassium cyanide solution and 150 ml buffer solution and dilute to 600 ml with water. Add 5 drops of eriochrome black-T indicator and sufficient formaldehyde to just give a red colour.

6.3.4 Titrate slowly with EDTA solution to a blue green end point. Add 5 ml of formaldehyde, and if the blue green colour changes to red, titrate again with EDTA solution to blue green end point. Continue with the formaldehyde additions and EDTA titrations till the addition of formaldehyde has no effect within 2 minutes of the blue green end point.

6.4 Calculation

$$\text{Zinc, percent} = \frac{A \times B}{C} \times 100$$

where

A = ml of EDTA solution required for titration of the solution,

B = EDTA equivalent in terms of grams of zinc per ml, and

C = mass in g of the sample represented in the aliquot used.

INDIAN STANDARDS

ON

PRECIOUS METALS

IS:

- 639-1965 Gold leaf (*revised*)
- 1417-1971 Grades of gold and gold alloys (*first revision*)
- 1418-1972 Method for assaying of gold in gold and gold alloys
- 1953-1973 Methods of chemical analysis of silver anodes (*first revision*)
- 2112-1962 Grades of silver and silver alloys
- 2113-1972 Methods for assaying of silver in silver and silver alloys (*first revision*)
- 2270-1965 Methods for assaying of platinum and platinum alloys
- 2271-1967 Recommended method for spectrographic analysis of platinum
- 2275-1963 Grades of platinum
- 2278-1963 Fine gold bar, sheet, wire, granules and token (*LAGDI or MOHUR*)
- 2279-1963 Fine silver bar, sheet, wire, granules and token (*LAGDI or MOHUR*)
- 2767-1964 Gold thread (silver base)
- 2790-1964 14, 12 and 9 carat gold
- 3088-1967 Method for assaying of fine grade palladium
- 3095-1965 Solders for use in goldwares
- 3096-1965 Fine grade palladium
- 3110-1965 Silver leaf
- 3111-1965 Silver thread
- 3112-1965 Gold and silver embroidery materials
- 3541-1966 Code of practice for manufacture of 14 and lower carat gold alloys
- 3571-1966 Dental gold solders
- 3578-1966 Dental gold alloy wire
- 3610-1966 Dental gold foil
- 4134-1967 Recommended colour classification of rough diamond
- 4704-1968 Silver-tin dental amalgam alloy
- 4705-1968 Dental mercury
- 4799-1968 Dental casting gold alloys
- 5320-1969 Fine silver ingot
- 5954-1970 Dental white gold alloys
- 6018-1971 Platinum, platinum-rhodium catalyst gauzes
- 6019-1971 Platinum dish
- 6170-1971 Platinum crucible and lid
- 6683-1973 Wires of platinum group metal and their alloys
- 6882-1973 Platinum electrodes
- 6889-1973 Methods for chemical analysis of silver-tin dental amalgam alloy
- 6890 (Part I)-1973 Methods for chemical analysis of dental gold alloys: Part I Determination of gold, silver, copper, palladium and platinum
- 7225-1974 Dental cobalt-chromium casting alloy
- 7255 (Part I)-1974 Methods for chemical analysis of solders for use in goldware: Part I Determination of gold, silver and copper
- 7562-1974 Gold cladding

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